CUBoulder Organic Chemistry Undergraduate Courses

Lab Techniques

Solvent Removal

It is often necessary to remove solvent from a solution to recover either a solid or a high-boiling liquid. There are several ways to do this.

Distillation

Simple distillation can be used to remove solvent. Distillation works well if the solution is composed of a solid and a low-boiling solvent, or if the solution is composed of a high-boiling liquid and a low-boiling solvent (with boiling point differences greater than 100°). Advantages of distillation are that the solvent can be collected and recycled and that no vapors are released into the atmosphere. A disadvantage is that it can take a long time. Simple distillation is covered on the simple distillation web page.

Open-Dish Evaporation

Solvent can be evaporated by placing the solution in an open container (an Erlenmeyer, evaporating dish, beaker, vial). The container is set on a heat source (steam bath, hot plate, heating mantle, sand bath) and the solvent boiled off. (If the solvent is water, use a heat source other than a steam bath.)

The problem with open-dish evaporation is that the solvent is released into the air. Open-dish evaporation should always be done in a hood if the solvent is anything other than water. Even in a hood, however, vapors are released into *somebody's* air. If the solvent is a hazardous compound (for instance, methylene chloride), it is probably better to choose another method of solvent removal.

Reduced-Pressure Evaporation

You can accomplish evaporation from a solution quickly by placing it in a side-arm flask, sealing the flask, and then applying vacuum. Under vacuum - reduced pressure - liquids vaporize and boil off at lower temperatures; effectively, the solvents come off a lot faster when under vacuum than at atmospheric pressure.

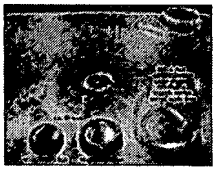
In the Organic Chemistry Teaching Labs, a small (25 or 50 mL) side-arm flask fitted with a rubber stopper is used to strip off small (5-10 mL) amounts of solvent. As a vacuum source, we use the VacuuBrand systems. Details follow.

Procedure

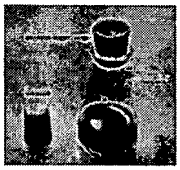
(click on the thumbnails to see a larger image)

Put the solution in a 25 or 50 mL side-arm flask. Do not fill the flask more than

one-third full, since the evaporation causes the solvent to froth and bubble up and out of the flask.

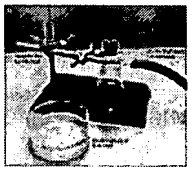


A boiling chip is not necessary, but can be helpful, especially if you do not plan to hold and swirl the flask during the process.



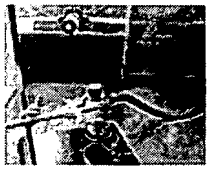
Stopper the flask with a black stopper. (Corks do not give a good seal.)

Clamp the flask to a ring stand to prevent it from falling over.

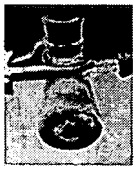


Connect the flask with vacuum tubing to the VacuuBrand vacuum source. Do NOT use Tygon tubing.

Have ready a small dish containing warm water.



Turn on the vacuum.



The solution will bubble and froth, especially when you first turn on the vacuum.

The flask will become cool as the solvent evaporates - place it in the warm water bath to speed up the evaporation.

You can remove the flask from the clamp and hold it in your hand and constantly swirl it during the process both to prevent bumping and to increase the surface area to speed up the process.

You are done when all the solvent has evaporated.

First turn off the vacuum source connection at your lab bench,

and **then** disconnect the flask from the vacuum tubing.

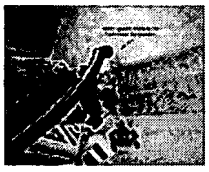
Rotary Evaporators

Rotary evaporators, or roto-vaps, are standard equipment in most organic chemistry research labs. These evaporators are designed to remove solvent rapidly from solutions. In the Organic Chemistry Teaching Labs at CU Boulder, we have about 6 roto-vaps on carts so that we can move them to where they are needed.

Procedure

First: look at the roto-vap. The motor in the roto-vap turns the flask rapidly, providing a greater surface from which evaporation can occur, thus speeding up the process. Cooling coils in the roto-vap condense the vapors and drop them into a collection flask so that they can be recycled or properly disposed. The roto-vap is connected to a vacuum source, again, this speeds up the evaporation process.

The picture to the right is a close-up of the tubing connected to the roto-vap.



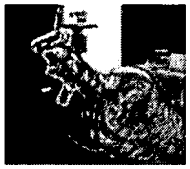
There are three tubing outlets on the roto-vap, one for a vacuum source and two for the cooling coils. Use vacuum tubing to connect the outlet that evacuates the roto-vap chamber to the vacuum source. Use a piece of Tygon tubing to connect one cooling coil outlet to the cold water faucet, and use another piece of Tygon tubing to connect the other cooling coil outlet to the drain.

Place the solution to be evaporated in a round bottom flask, then connect the flask to the roto-vap. Do not fill the flask more than about one-third full.

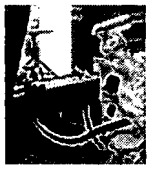
Use a joint clamp to secure the flask to the apparatus. Make sure the cool water to the cooling coils is turned on.

Turn on the motor so that the flask rotates.

Usually the flask containing the solution to be evaporated is warmed by a water bath; consult your TA to see if it is necessary.



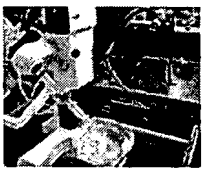
Make sure the vent at the top of the cooling coils is closed.



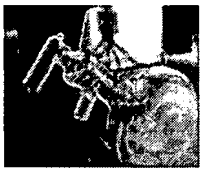
Turn the vacuum outlet on the vacuum system on.

As the solvent evaporates, you may notice a lot of frothing and bubbling in the evaporating flask. If it starts bubbling out of the flask, you can open the vent a little (see picture above) to release some of the pressure.

The (unwanted) solvent condenses on the cooling coils and drips down into the collection flask.



When the solvent has evaporated, turn off the motor that turns the flask and turn the vacuum outlet to closed.



Slowly open the vent to release the pressure in the roto-vap chamber.

Remove the flask from the roto-vap.

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